

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.**

(19) **FEDERAL  
REPUBLIC OF  
GERMANY**

**GERMAN PATENT  
OFFICE**

(12) **Unexamined Patent  
Application**

(10) **DE 41 04 513 A1**

(21) File no.: P 41 04 513.0

(22) Application date: 14.2.91

(43) Date laid open  
for public inspection: 29.8.91

(51) Int. Cl. 5:  
**B 01 J 20/34**  
B 01 J 20/20  
B 01 J 20/28  
C 01 B 31/08  
B 01 D 53/02  
B 01 D 61/00  
B 01 D 69/00  
// C02F 1/28

(30) Internal priority: (32) (33) (31)  
14.02.90 DE 40 04 533.1 27.12.90  
DE 40 41 911.8

(71) Applicant:  
Chmiel, Horst, Prof. Dr-Ing.habil., 7250  
Leonberg, DE

(74) Representative:  
Munich, W., Dipl.-Phys.Dr.rer.nat., Pat.-  
Att.; Steinmann, O., Dr., Attorney-at-law,  
8000 Munich

(72) Inventor:  
same as applicant

(54) Process for the regeneration of adsorbers  
(57) A process for the regeneration of adsorbers is  
described, in which the adsorbing material is  
electrically conductive and is heated using  
electric current to a temperature at which  
the adsorbed material is expelled.  
The process according to the invention is  
characterized in that compressed or fibrous  
activated charcoal is used as adsorbing  
electrically conductive material, and the  
current passage is such that the whole  
volume of the material is uniformly  
heated.

## Description

The invention relates to a process for the regeneration of adsorbers, in  
5 which the adsorbing material (adsorbent) is heated to a temperature at which  
the adsorbed material is expelled, i.e. desorbed.

In recent years adsorbers have found increased use inter alia for the  
removal of pollutants from water or air. For example, chlorinated  
hydrocarbons (CHCs) can be removed even in low concentrations by means  
10 of adsorbers from fluid flows, such as flows of gas or water. A typical use is  
the redevelopment of abandoned polluted sites contaminated by CHCs by  
suction with integrated adsorption of the pollutants. Activated charcoal is often  
used as adsorber.

As the adsorptive capacity declines somewhat due to the "occupancy"  
15 of the adsorber spaces, it is necessary to regenerate the adsorbing material  
by expelling the adsorbing material. However this regeneration often causes  
problems:

At present the so-called displacement process using steam is most  
often used; in this process the regeneration of for example activated charcoal  
20 is only partially successful, so that, after several regeneration cycles, the  
efficiency of the adsorbing material has declined to the point where it must be  
disposed of at great expense.

W. Kast therefore suggests in his book "Adsorption aus der Gasphase"  
[Adsorption from the gas phase] (Verlag VCH Weinheim, 1988) the thermal  
25 regeneration or the combination of thermal regeneration and the displacement  
process. To carry out this regeneration process the activated charcoal must  
however be heated over a heat exchanger. In most cases this means that the  
activated charcoal has to be removed from the adsorption device before  
regeneration as, due to its poor heat transfer, a heating of the adsorber  
30 column from outside is uneconomical.

Furthermore a process and a device for the regeneration of activated  
charcoal are known from DE 29 53 672 A1 in which an arc which is generated  
by a pulsating voltage releases the adsorbed substance. The use of arcs

leads however to pronounced burning-off and thus to a rapid consumption or wear of the adsorber.

Furthermore a process is known from US-PS 42 61 857 in which consumed activated charcoal is poured into a hermetically sealed oven with  
5 several electrodes arranged vertically at intervals. An electric current which heats the activated charcoal is passed over the electrodes so that the adsorbed substances are released.

In this process and also that known from US-PS 42 61 857 the activated charcoal must be present in "particle form". The use of powdery or  
10 granular activated charcoal has however the disadvantage that both the electric and the heat conduction resistance is high so that the regeneration efficiency is low. In addition, due to the non-uniform charging, hot spots form during the electric heating of particulate, swirtable activated charcoal, which can lead to spontaneous ignition upon subsequent re-use as adsorber.

Moreover it is necessary in the case of the known processes and  
15 devices in which the activated charcoal is electrically heated to remove the activated charcoal from the actual adsorption device for the purpose of regeneration.

The object of the invention is to provide a process for the regeneration  
20 of adsorbers consisting of activated charcoal which can be carried out without having to remove the adsorbing material from the adsorption device, and which has a high regeneration efficiency without hot spots being able to form.

The achievement of this object, according to the invention, is given in claim 1. Further developments of the invention are the subject-matter of the  
25 dependent claims.

This object is achieved according to the invention in that the starting-point is a process for the regeneration of adsorbers in which the adsorbing material (activated charcoal) is heated to a temperature at which the adsorbed material is expelled, i.e. desorbed.

According to the invention an electrically conductive material is used as  
30 adsorbing material, the activated charcoal being made uniformly electrically conductive for example by being compressed and sintered as tube, an adequate electrical conductivity being maintained given suitable preparation.

To heat the electrically conductive adsorbing material, it is heated by current passage, such that the adsorbing material is expelled.

The heating can take place by direct current passage (claim 3). Suitable electrodes are provided for this, to which a direct or alternating  
5 voltage is applied.

Furthermore it is also possible to heat the electrically conductive adsorbing material inductively with a known induction heating device (claim 4).

Moreover it is however also possible, instead of or in addition to heating by current passage, to carry out the heating of the adsorbing material  
10 by a microwave heater by which the whole volume of the material is likewise uniformly heated (claim 5).

In each case the regeneration of the adsorbing material can take place both in the actual adsorption device and outside the adsorption device. In the case of a (as a rule preferred) regeneration of the adsorbing material in the  
15 adsorption device it is of course to be ensured that the desorbed pollutants are collected in suitable manner.

The process according to the invention in which a heating of the adsorbing material takes place by current passage and/or microwave heating can be used regardless of the structural form of the adsorber: thus it is  
20 possible to use as adsorbers hollow fibres or hollow columns through which the polluted medium flows, or to use mesh-shaped structures.

In the case of the use of activated charcoal as adsorbing material the activated charcoal can easily be prepared in the desired geometric form by coking of suitable structures. There can be used as starting materials e.g.  
25 briquettes made from charcoal, extruded pitch or any polymers such as nylon, polyamides, cellulose etc which are present in the desired form and which are heated to the coking temperature with exclusion of air. Upon reaching a specific degree of coking an electrical conductivity results which allows an adequate heating of adsorbers with customary voltages.

30 In each case it is however an advantage if the pollutants desorbed during the regeneration phase are removed by a carrier-gas stream. The carrier-gas stream can be for example an inert gas (claim 8) or steam (claim 7). When steam is used as carrier gas the thermal regeneration and the displacement regeneration are combined with each other on the one hand and

on the other hand the pollutants are separated particularly easily by condensation. However the direct heating prevents the formation of steam condensate in the pores of the adsorbing material which, with the known displacement regeneration processes, hinders the diffusion of the desorbent pollutant molecules and thus ends the regeneration after a few cycles.

The invention is described in more detail below with reference to the drawing, in which are shown:

Figs. 1 to 4 various embodiments of the invention.

Figs. 1 to 4 show adsorber devices which are designed according to the invention such that they make possible an "in situ" regeneration of the adsorbing material. To this end, the adsorbing material is present in the form of hollow tubes 2 which have been made by a compression or sintering process.

In order to make possible a heating of the adsorbing material for example by direct current passage, suitable electrodes 1' and 1'' are provided in the embodiments shown, to which a direct or alternating voltage is applied. Furthermore it is also possible to heat the electrically conductive adsorbing material 2 inductively with a known induction heating device.

Fig. 1 shows the basic structure of a device in which tubes 2 are used as adsorbing material, while Fig. 2 shows a possibility of the contacting of the tubes 2 by the electrodes 1: By pressing the electrode 1' towards the tubes 2 by means of a spring 3, a particularly small contact resistance results.

Fig. 3 shows an embodiment in which the adsorbing material is present in the form of activated charcoal capillaries 2' into which for example solvent-loaded air enters, which then emerges as solvent-free air. The capillaries 2' are connected by means of a conductive adhesive 4 to a metal ring 5 made of Al, Cu or VA, which serves as electrode.

In every case the regeneration of the adsorbing material 2 can be carried out both in the actual adsorption device and outside the adsorption device. During a regeneration of the adsorbing material in the adsorption device it is of course to be ensured that the desorbed pollutants are collected in suitable way.

This can take place immediately by applying a vacuum with adjacent cooling trap or by inserting a solvent-selective membrane according to Fig. 4

in the embodiment shown which is a solvent-selective polymer film 6 which is applied directly to the tube 2 made of activated charcoal.

5 The embodiments shown can be easily produced for example by coking a material present in the desired geometric form. There can be used as starting material e.g. briquettes made from charcoal, extruded pitch or any polymers such as nylon, polyamides, cellulose etc, which are present in the desired form and which can be heated to the coking temperature with exclusion of air. Upon reaching a specific degree of coking an electrical conductivity results which allows an adequate heating of adsorbers with  
10 customary voltages.

Under certain circumstances it is advantageous if the pollutants desorbed during the regeneration phase are removed by a carrier-gas stream. The carrier-gas stream can be for example an inert gas or steam. When steam is used as carrier gas the thermal regeneration and the displacement  
15 regeneration are combined with each other on the one hand and on the other hand the pollutants are separated particularly easily by condensation. However, the direct heating prevents the formation of steam condensate in the pores of the adsorbing material which, with the known displacement regeneration processes, hinders the diffusion of the desorbent pollutant  
20 molecules and thus ends the regeneration after a few cycles.

When carrying out the process according to the invention it is furthermore advantageous if two or more adsorber columns are used the adsorbing material of which is electrically conductive. By using at least two adsorber columns the device can be alternately loaded and regenerated by  
25 simple switching of one or more columns so that a continuous operation of the adsorption device is possible without changing the adsorber.

## Patent claims

1. Process for the regeneration of adsorbers in which the adsorbing material is electrically conductive and is heated using electric current to a temperature at which the adsorbing material is expelled, **characterized in that** compressed or fibrous activated charcoal is used as adsorbent electrically conductive material and that the current passage takes place such that the whole volume of the material is uniformly heated.
2. Process according to claim 1, characterized in that there is used as adsorbing material activated charcoal which is made uniformly electrically conductive by sintering.
3. Process according to claims 1 and 2, characterized in that the adsorbing material is heated by direct current passage.
4. Process according to claims 1 and 2, characterized in that the adsorbing material is inductively heated with an induction heater.
5. Process for the regeneration of adsorbers, in which the adsorbing material is heated to a temperature at which the adsorbing material is expelled, characterized in that compressed or fibrous activated charcoal is used as adsorbing material and the heating takes place by a microwave heater such that the whole volume of the material is uniformly heated.
6. Process according to one of claims 1 to 5, characterized in that the regeneration takes place in the adsorber device.
7. Process according to one of claims 1 to 6, characterized in that steam is used as flushing gas.
8. Process according to one of claims 1 to 7, characterized in that an inert gas is used as flushing gas.
9. Process according to one of claims 1 to 8, characterized in that at least two adsorber columns are used which are alternately loaded with the polluted fluid or regenerated.
10. Device for carrying out the process according to one of claims 1 to 9, characterized in that the adsorbing material is present in the form of tubes or hollow fibres, through which the polluted fluid flows.



11. Device for carrying out the process according to one of claims 1 to 9, characterized in that the adsorbing material is present in the form of mats through which the polluted fluid flows.
  12. Device according to claim 10 or 11, characterized in that a solvent-selective membrane is provided in which the desorbed solvent preferably dissolves.
- 5

4 pages of drawings

Number: DE 41 04 513 A1  
Int. Cl.<sup>5</sup>: B 01 J 20/34  
Date laid open  
for public inspection: 29<sup>th</sup> August 1991

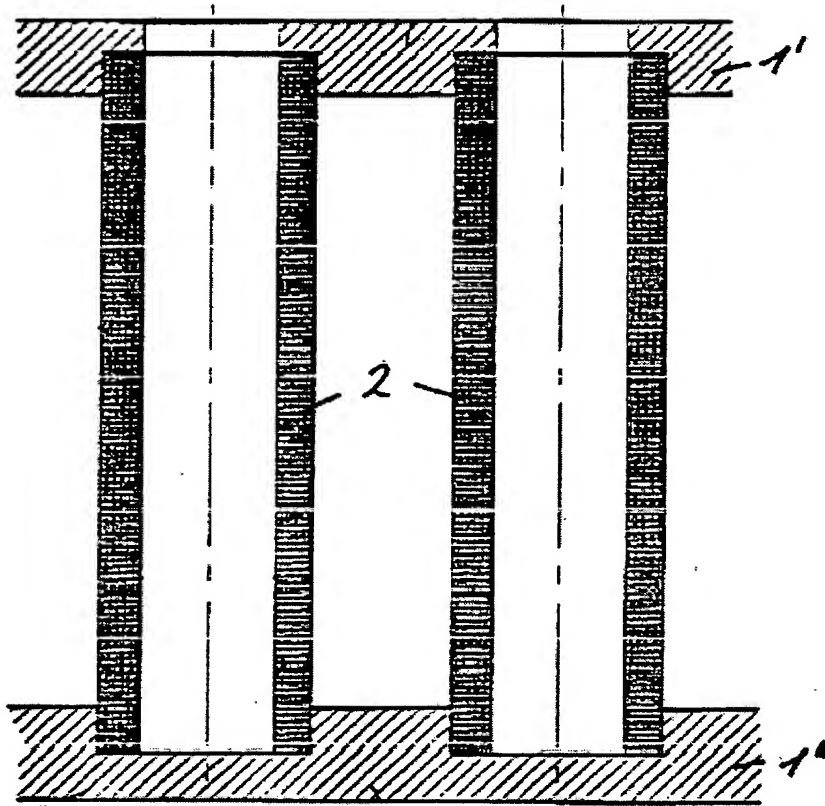


Fig. 1

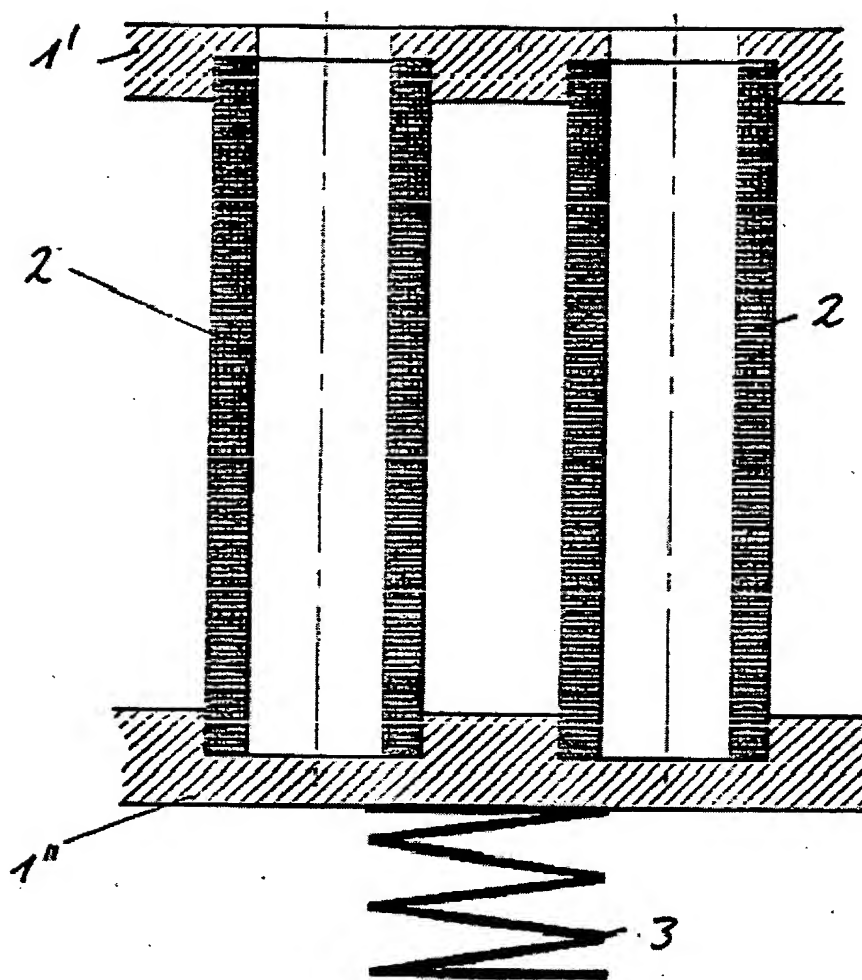


Fig. 2

Number: DE 41 04 513 A1  
Int. Cl.<sup>5</sup>: B 01 J 20/34  
Date laid open  
for public inspection: 29<sup>th</sup> August 1991

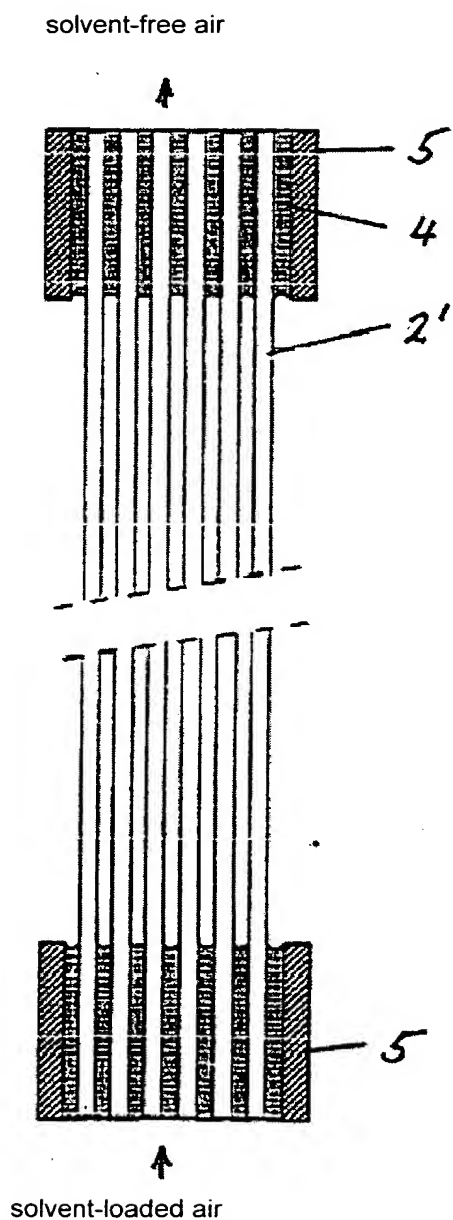


Fig. 3

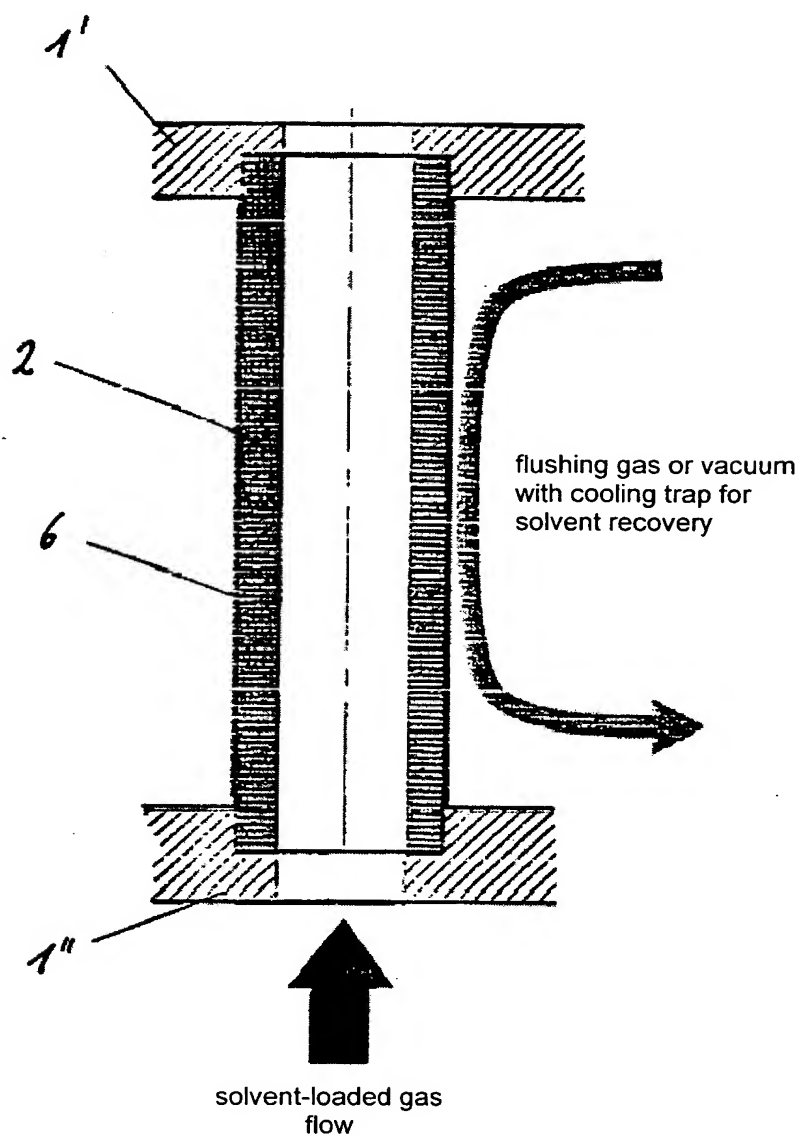


Fig. 4